6-15-2004

Re-examination of "missing strain" during superplastic deformation of AA7475

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Published in *Materials Science and Engineering A* **374**:1-2 (June 15, 2004), pp. 369-373; doi: 10.1016/j.msea.2004.03.034

Submitted July 7, 2003; revised March 8, 2004; published online May 18, 2004.

Re-examination of "missing strain" during superplastic deformation of AA7475

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Abstract

"Missing strain," a discrepancy between the total macroscopic strain and the strain contributed by grain boundary sliding (GBS) during superplastic deformation, appears to exist in previous investigations. In this work, the contribution of GBS (ξ_{GBS}) and intragranular strain (ξ_{IG}) were simultaneously measured using scratch test conducted on AA7475 samples after deformation at 500 °C at initial strain rate of 10^{-3} s^{-1}. The result shows that the missing strain results most probably from the neglect of the intragranular strain as well as the anisotropic GBS-induced underestimation of ξ_{GBS}. The calculation of ξ_{GBS} was re-examined, based on the fact that anisotropic shrinkage of samples along width and thickness during superplastic deformation was noted.

Keywords: missing strain, grain boundary sliding, superplastic deformation

1. Introduction

The superplastic behavior of materials is attributed to many different mechanisms including grain boundary sliding (GBS), intragranular strain, diffusional flow, grain rotation, cavitation, etc. GBS has been shown to be the largest contributor to the total strain. Earlier surface marker experimental results have shown that the scratch remains straight inside grains and grain elongation is absent after superplastic flow, which indicates that intragranular deformation contribution to optimal superplastic flow is negligible [1, 2]. On the contrary, the curvature of scratches within a grain also has been reported [3], suggesting that intragranular deformation is also present.

The contribution of GBS to total strain, ξ_{GBS}, the ratio of ε_{GBS} to the total strain, is usually measured using the method Langdon proposed [2 and 4]. When two grains move over each other in a polycrystalline materials, with the displacement taking place along their mutual boundary, the sliding vector may be resolved into three mutually perpendicular components, as illustrated in Figure 1.

In practice, measuring the separations between the end points of a broken marker line is usually difficult. Therefore, determinations of ε_{GBS} relied upon taking measurements of the w component of sliding, instead of u, and calculating ε_{GBS} from the relationship:

$$\varepsilon_{GBS} = \frac{1}{\bar{L}_1}\overline{\left(\frac{v}{\tan\varphi} + \frac{w}{\tan\theta}\right)_1} = \frac{2}{\bar{L}_1}\overline{\left(\frac{w}{\tan\theta}\right)_1} = \Phi\frac{\bar{w}_1}{\bar{L}_1} \quad (1)$$

where Φ is a constant. Meanings of w, v, φ, θ are indicated in Figure 1. $\bar{L}$ is the mean linear intercept grain size and subscript l denotes the procedure of taking measurements along a longitudinal traverse. Langdon [2, 4] recommended the use of Φ = 1.5 since value of Φ was estimated to be approximately 1.62 from the theoretical distribution of w versus θ [5] and about 1.44 from experiment. In Equation (1), it was assumed that v and w have no distinct physical difference in the sample interior, i.e.

$$\overline{\left(\frac{v}{\tan\varphi}\right)_1} = \overline{\left(\frac{w}{\tan\theta}\right)_1} \quad (2)$$

The measured ξ_{GBS} never approaches 100%, although under optimum superplastic condition GBS is considered to account for all the deformation. "Missing strain" [1, 2], a

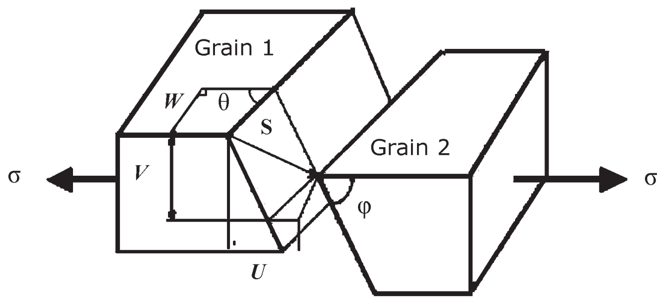

Figure 1. Grain boundary sliding between grains 1 and 2 showing the three components of sliding, namely *u*, *v*, and *w*.

discrepancy between total macroscopic strain and strain contributed by GBS, appears to exist in those previous investigations. Many experiments have showed ξ_{GBS} lying in the range of 50–70% [6–11]. Langdon [2] suggested that, depending upon the precise mechanism of accommodative sliding, the experimental method used to measure sliding will lead to values of ξ_{GBS} in the range of 45–90% even when grain boundary sliding accounts for all the deformation [2]. Thus, the discrepancy most probably results from the experimental limitations of the GBS measurement.

Besides the measurement limitations, the missing strain is suspected to result from intragranular strain that is ignored, since grain elongation along tensile direction has been reported [12]. In this work, GBS and intragranular strain contributions were measured, and at the same time the experimental limitations of GBS measurements was reexamined, aiming to clarify the source of this discrepancy.

2. Experimental

The material used is AA7475. The microstructure and superplastic behavior of the material can be found in [13]. The average linear intercepts of grains of the material are 10, 10.1 and 7.2 μm in rolling (R), transverse (T) and short transverse (ST) directions, respectively. I-type tensile samples were cut from AA7475 sheet, with the sample's length parallel to the rolling direction of the sheet.

The material was found to show superplastic behavior at test temperature ranging from 500 to 530 °C and constant initial strain rate range from 3.3×10^{-4} to 10^{-3} s^{-1}. The selection of the test parameters for GBS determination has taken into consideration of grain growth during the high temperature exposure. It is found that, during the short exposure (equivalent to the time needed for a complete thermal cycle of superplastic deformation to 100% strain at 10^{-3} s^{-1}) at high temperature, the static grain growth can be ignored even for the sample which has been exposed to 530 °C. The retardant static grain growth is attributed to the presence of precipitates on the grain boundaries, which were induced during thermal mechanical processing (TMP) of the material. However, with the presence of stress at high temperature, grain growth is notable especially at 516 and 530 °C. Therefore, the current study was conducted at 10^{-3} s^{-1} at 500 °C, when the average grain growth of materials after strained to 100% is only about 10%. Thus, the GBS measurement could be conducted with relatively good certainty. This is also the reason why the optimum test condition of the material AA7475, 10^{-3} s^{-1} and 516 °C, was not used for this study, and different from that of our previous studies in [13, 14]. The elongation to failure of the material for the chosen parameters is 690±10%.

In order to induce surface markers, samples were polished up to 0.05 μm colloid silica slurry. Marker lines parallel or perpendicular to the tensile axis were then placed on the polished surface of the samples using 1 μm diamond paste and a lens tissue. The measurement of GBS contribution (ξ_{GBS}) and intragranular strain contribution (ξ_{IG}) were performed on one of the R–T faces. It is relatively easy for these measurements and observations to be conducted on the R–T faces, compared with that on T–ST faces, due to the geometry of the sample. The two T–ST surfaces were polished using 1 μm diamond paste manually in order to reduce possible surface defects.

The measurement of ξ_{GBS} was carried out through the measurement of surface scratch offset along sample's width direction before and after superplastic deformation (w) as shown in Figure 1, with the aid of SEM observation. ε_{GBS} then was calculated using Equation (1). The variations of the distance between the two parallel scratches inside grains which had been placed in a perpendicular direction to the tensile axis were used to determine the intragranular strain. Intragranular strain (ε_{IG}') of a single grain is calculated with

$$\varepsilon'_{IG} = \frac{l_t - l_0}{l_0} \tag{3}$$

where l_t, l_0 are the distance between two marker lines inside grain after and before the deformation, respectively. In this study, ε_{GBS} and ε_{IG} were calculated by averaging of about 40–50 measurements and then ξ_{GBS} and ξ_{IG} were calculated as the ratio of ε_{GBS} and ε_{IG} to the total strain. Typical morphologies of the sample surface for ε_{IG} determination are showed in Figure 2.

Contribution of diffusional creep to the total strain was also estimated in this work. It has been suggested that the contribution of diffusional creep could be determined by the measurement of the size of the precipitate free zone (DFZ) of Al alloys [15]. DFZ was also observed in this work. However, accurate measurement of DFZ size was rather difficult, since in most cases the fringes of the DFZ were curved and unclear. The contribution of the diffusional creep then was estimated by the measurement of DFZ area with the aid of image analyzer. If the DFZ resulted from the diffusional creep and it fully contributes to total strain, the contribution of diffusional creep is equal to the ratio of the area of DFZ to the total grain area:

$$\varepsilon_{DC} = \frac{S_{DFZ}}{S_{TOTAL}} \tag{4}$$

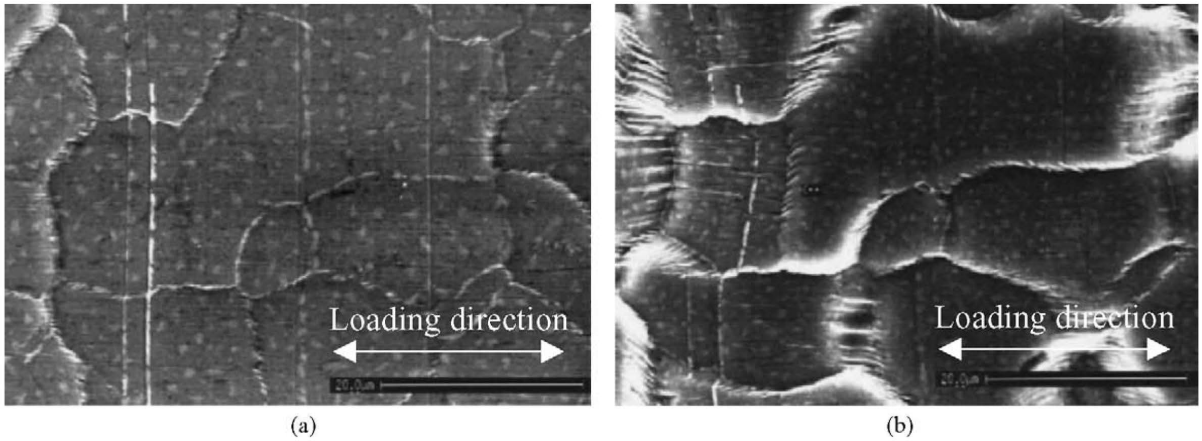

Figure 2. Comparison of SEM morphologies of sample surface at (a) ε = 20% and (b) ε = 50% reveals the presence of GBS and intragranular strain. Sample was deformed at 500 °C at initial strain rate 10^{-3} s^{-1}, stressed horizontally.

where S_{DFZ} and S_{TOTAL} are the total area of DFZ and total area of grain, respectively. Contribution of diffusional creep (ξ_{DC}) can then be determined by the ratio of ε_{DC} to the total strain.

3. Results and discussion

3.1. Contribution of intragranular strain (ξ_{IG})

In this investigation, measurements of ξ_{GBS} and ξ_{IG} were carried out simultaneously. The results are presented in Figure 3. ξ_{GBS} is in the range of 51–65% and ξ_{IG} is in the range of 33–19%. ξ_{GBS} is consistent with results reported in [7–11]. It is noted that ξ_{GBS} increases from 51 to 65% with the increase of strain from 0 to 200%. On the contrary, ξ_{IG} decreases from 33 to 19% as the strain is increased from 0 to 200%. This suggests that GBS may not fully act in the earlier stage of deformation, especially before the maximum stress. This behavior may be related to the evolution of the low-angle grain boundaries of the material with increasing strain [14].

The "missing strain" is checked by the examination of the presence of intragranular strain and diffusional flow. Langdon [1] showed that the intragranular strain is non-uniform; it tends to have oscillatory character with both positive and negative components, and it makes no net contribution to the total strain. This is because with the presence of the extensive grain rotation, the net contribution of intragranular strain would be offset by grain rotation. However, in the present test, other than the presence of intragranular strain in a narrow strain range (when grain rotation is limited) as shown in Figure 3, grain elongation along the tensile direction in the whole strain range (when grain rotation is extensive) was observed, suggesting that the intragranular strain cannot be fully offset by grain rotation. As can be seen in Figure 4, the length-to-width ratio of grain, (R), the ratio between $\overline{L}_{//}$ and $\overline{L}_{\perp}$, reveals the presence of the plastic deformation of grains, especially at earlier stage of deformation. $\overline{L}_{//}$ and $\overline{L}_{\perp}$ are the linear intercept of grains measured along directions parallel and perpendicular to the tensile directions, respectively.

The variation of grain shape provides another way to evaluate the net contribution of intragranular grain. If the

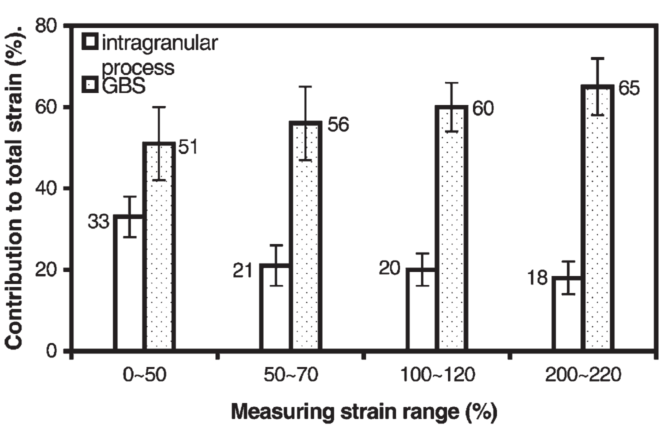

Figure 3. Contributions of GBS and intragranular strain to the total strain at different strain ranges, at 500 °C, at initial strain rate 10^{-3} s^{-1}.

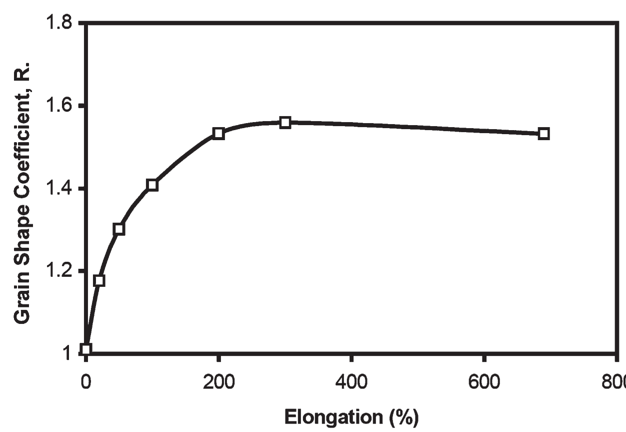

Figure 4. Variation of length-to-width ratio of grain (R) as a function of elongation, at 500 °C, at initial strain rate 10^{-3} s^{-1}.

Table 1. List of shrinkage (%) of sample dimensions along width and thickness of the sample after superplastic deformation

	Strain			
	50%	100%	200%	690%
Thickness shrinkage, S_t (%)	23 ±1	34 ± 2	45 ± 2	65 ± 4
Width shrinkage, S_w (%)	14 ±1	22 ± 2	37 ± 2	57 ± 3
Ratio S_t/S_w, α	1.64	1.55	1.22	1.14

volume of the material remains constant during the deformation, and the shrinkages in both transverse and short transverse directions are uniform when the material is elongated along the longitudinal direction, the relationship between the strain and length-to-width ratio of grain can be expressed simply as following:

$$\frac{R_t}{R_0} = (1+\varepsilon)^{2/3} \tag{5}$$

where ε is the plastic deformation the grain experienced and R_t and R_0 are the length-to-width ratio of grain after and before deformation, respectively. ε then are estimated to be 19, 26 and 33%; and their corresponding ξ_{IG} are 38, 26 and 16.5%, at total strain of 50, 100 and 200%, respectively. ξ_{IG} might be overestimated here as diffusional flow can contribute to the increasing of $\bar{L}_{//}$ and decreasing of $\bar{L}_{\perp}$. Nevertheless, this is in good agreement with intragranular strain that has been measured using the scratch test.

The total contribution of GBS and intragranular strain to the total strain is determined to be about 80%. Thus, contribution of intragranular strain can be an important contributor but cannot account for all the "missing strain". There is still a "missing strain" of about 15% of the total strain, even with the consideration of ξ_{IG} and ξ_{DC}. The latter is estimated to be in range of 1–7% in the strain range of 0–200%.

3.2. *Re-examination of ξ_{GBS}*

As ξ_{IG} and ξ_{DC} cannot fully account for the "missing strain", it is therefore necessary to look into the procedure for determining of ξ_{GBS}. It is noted that the contraction of sample gauge along width and thickness direction of the sample after deformation is anisotropic. The shrinkages of samples along width and thickness are listed in Table 1. As can be seen from Table 1, shrinkage of the sample thickness is more significant than that of sample width, especially at the low strain range. The ratios between them (α) range from 1.64 to 1.14. α decreases with the increasing of strain from 50 to 660%.

The evolution of the grain shape (intragranular strain) and GBS should commensurate with the change of specimen shape. According to above calculation in Section 3.1, it implies that the grain shrinkage along T and ST direction could be at a similar ratio. The anisotropic shrinkage along width and thickness of the sample, therefore, indicates that the grain boundary sliding could be anisotropic. Thus, Equation (4) needs to be re-examined. As mentioned above, the reduction from (2), (3), and (4) is based on the assumption, $\overline{(v/\tan\varphi)_1} = \overline{(v/\tan\theta)_1}$, when v and w have no distinct physical difference in the sample interior. In this investigation, shrinkage along sample thickness, contributed by v, is found to be more significant than shrinkage along sample width, which is contributed by w (see Figure 1). v is determined to be α times larger than w, i.e. $v = \alpha w$, and α > 1, as shown in Table 1. Thus, Equation (3) is changed to

$$\overline{\left(\frac{v}{\tan\varphi}\right)_1} = \alpha\overline{\left(\frac{w}{\tan\varphi}\right)_1} = \alpha\overline{\left(\frac{w}{\tan\theta}\right)_1} \tag{6}$$

as there is no difference between θ and φ statistically. If the theoretical distribution of w versus θ keeps [1, 5] and Φ is taken as usual (Φ=1.5), Equation (4) then can be rewritten as

$$\varepsilon_{\text{GBS}} = \frac{1+\alpha}{2}\Phi\frac{\overline{w}_1}{\bar{L}_1} = \beta\Phi\frac{\overline{w}_1}{\bar{L}_1} \tag{7}$$

Here, due to the anisotropic shrinkage ratio of the sample, a modification coefficient β is needed for the calculation of strain contributed by GBS. β can be calculated from Table 1. The values of β and the recalculated results of GBS contribution to the total strain are presented in Table 2.

The GBS contribution after correction is now in the range of 67–76%, instead of 51–65%. The total contribution from GBS and intragranular strain now is approaching 100%. If there is presence of diffusional flow to the total strain, $\xi_{GBS} + \xi_{IG} + \xi_{DF}$ is very close to unity. However, this correction could be underestimated or overestimated, as the shrinkage of the sample dimension can be partially contributed by the possible anisotropic shrinkage of grains under stress. This is worthy of further studies.

Langdon re-examined the "missing strain" in [1, 2]. It is concluded that the experimental method used to measure sliding will lead to values of ξ_{GBS} in the range of 45–90%, even when grain boundary sliding accounts for all the de-

Table 2. Re-examination of ξ_{GBS}

	Strain				
	20–50%	50–70%	100–120%	200–220%	660%
Ratio S_t/S_w, α	1.64	1.64	1.55	1.22	1.14
Correction coefficient, β	1.32	1.32	1.27	1.11	1.07
$\xi_{GBS} = (\beta\Phi(\overline{w_1}/\overline{L_1}))/\varepsilon_{total}$	67	74	76	72	NA
$\xi_{GBS} + \xi_{IG}$	100	95	96	91	NA

formation. The experimental values of ξ_{GBS} = 50–70% underestimates the true values of the sliding contributions because of the limitation in the measuring procedure. In this study, due to the measurement difficulties of ε_{GBS} directly from the *u* components of sliding measured parallel to the tensile axis, ε_{GBS} determined through measuring *w* is underestimated due to the presence of anisotropic GBS. The contributions of GBS after correction are 1.1–1.3 times higher that the original values; but there is still a difference of 20–30% to the unit. This gap is filled by the presence of intragranular strain, as shown in Section 3.1. The contributions of intragranular strain might be overstressed here as the current test is conducted slightly off the optimum superplastic deformation condition of the material. Nevertheless, combining the contribution of GBS after correction and intragranular strain (and even diffusional flow) is very close to unity. Therefore, the "missing strain" observed in the present study is mainly caused by the presence of intragranular strain, as well as the anisotropy of GBS caused ξ_{GBS} underestimation.

4. Conclusions

"Missing strain" is re-examined through the simultaneous measurement of contribution from grain boundary sliding (ξ_{GBS}), intragranular strain (ξ_{IG}) and diffusional flow (ξ_{DF}), as well as a review of the procedure for ξ_{GBS} determination. The contribution of intragranular strain should not be ignored and ξ_{GBS} is underestimated due to the anisotropic grain boundary sliding. Correction of ξ_{GBS} can be performed based on the anisotropic shrinkage of sample dimensions. After correction of ξ_{GBS}, the sum of ξ_{GBS} and ξ_{IG} is close to unity.

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